

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : William J. Rigby, et al.  
Title : A PROCESS FOR MANUFACTURING POTASSIUM  
SULFATE FERTILIZER AND OTHER METAL SULFATES  
Serial No. : 09/100,994  
Filing Date : June 22, 1998  
Art Unit : 1754  
Examiner : Eileen E. Nave  
Date : January 13, 2000

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DECLARATION OF WILLIAM J. RIGBY

William J. Rigby states that if called to testify, he would state as follows:

1. I received a diploma in engineering from St. Francis Xavier University in 1951 and a Bachelor's degree in chemical engineering in 1953 from Nova Scotia Technical College of Halifax, Nova Scotia, Canada.
2. I have had forty-six (46) years of experience as a chemical process engineer beginning from 1953 to 1955 working for Canadian Industries, Ltd. as a process engineer. The business of the plant in which I worked was an ammunition and detonator plant and I investigated causes of misfires and developed unique methods of preventing moisture penetration of blasting caps to minimize misfires.
3. From 1955-1956, I was process engineer for Canadian Industries, Ltd. at Millhaven Ontario Canada, where I worked on the start-up of a Dacron polyester fiber

plant. Methanol, the by-product of the chemical synthesis part of this plant was required to be of high quality for the manufacture of Bakelite by the only primary customer for this product. Inadequate plant design mitigated this requirement. I ran numerous tests and developed processes and procedures to show that a methanol still as designed could not produce methanol of the quality required by the companies or primary customer and that major modifications were required.

4. Between 1956-1960, I was both a shift foreman and then an area supervisor for Allied Chemical Corporation at Syracuse, New York. There, I was responsible for operation of calcining sections of 2000 NT/day soda ash plant and for a dense ash plant. I initiated and conducted studies to improve the operation by replacing coal firing on rotary calciners with natural gas. I also prepared appropriation request for a major change in the process. After the change was effected, the Syracuse Soda Ash Plant capacity increased from 2000 NT/day to over 3000 NT/day. This very significant change permitted this plant to continue profitable operation for many years following the market introduction of natural soda ash from Wyoming.

5. In 1960-1961, I was a technical supervisor for Allied Chemical Corporation at the Amherstburg Ontario, Canada plant. At the Amherstburg, I created modifications to the absorption system of the Soda Ash Plant which increased plant capacity from 800 NT/day to 1000 NT/day.

6. Between 1962-1965, I was assistant plant manager at the Allied Chemical Corp. at Amherstburg Ontario, Canada. There, I was responsible for the operation of the entire plant which consisted of a soda ash and dense ash plant, calcium chloride

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plant, aluminum chloride plant, lime kilns and a large limestone quarry. During my tenure, a large chlorinated hydrocarbon refrigeration plant was built and placed on line. The calcium chloride plant process was completely revamped.

7. From 1965-1966, I was assistant to the Director of Operations for the Solvay Process Division of Allied Chemical in Syracuse, New York. There I worked on appropriation request for the entire division and assisted the Director of Operations on a variety of technical matters.

8. From 1966-1968, I was transferred to the Agricultural Chemical Division as manager of Southpoint, Ohio chemical plant. Because this was an older plant with serious labor and operating problems, I was charged with restructuring the plant operations, significantly reducing the work force and installing plant improvements which resulted in preventing a plant shutdown. The plant processes consisted of the production of ammonia, urea, nitric acid, ammonium nitrate, methanol, formaldehyde, urea/formaldehyde resins and melamine.

9. From 1968-1972, I was general manager of the Geismar, Louisiana, Chemical Complex. As general manager, I oversaw the largest, most modern complex owned by Allied Chemical Company. The processes consisted of a large modern ethylene plant of about 1 million pounds per day, a hydrofluoric acid plant, an aluminum fluoride plant, as well as the production of ammonia, urea, two nitric acid and nitrogen solutions. We also oversaw 1000 NT/day sulfuric plant, phosphoric acid plant, a super phosphoric acid and an ammonium phosphate plant. This was a green field installation with major start-up problems prior to and during my tenure.

10. From 1972-1973, I was Project Manager of CF Industries in Bartow, Florida. I was responsible for the construction of a ship loading facility, a sulfur trucking operation and major expansion of the plant city facility of CF Industries. I introduced the unique concept of wet rock grinding for this plant and the phosphate industry. This eliminated drying and storing phosphate pebble rock at the mine site permitting transport in open coal car type hopper cars to plant rather than covered hopper cars which were in tight supply, permitted open pile storage at plant site and significantly increased phosphoric acid plant capacity. This process modification has been adopted by the entire phosphoric acid industry. I also initiated construction of two 1000 TPD sulfuric acid plants and 3 train DAP/granular triple super phosphate (TSP) plant.

11. From 1973-1974, I was general manager of all southern operations for CF Industries at Bartow, Florida. There, I was general manager of all Florida operations including plants that produced phosphoric acid, sulfuric acid, DAP and GTSP as well as an animal feed plants. I initiated sulfur trucking operations and supervised the operation of the port facility. At the time, this combination was the largest phosphate operation in the United States.

12. From 1974-1978, I was vice-president of operations for Nipro, Inc. & Columbia Nitrogen Corp. Both of these companies were the U.S. subsidiaries of the Dutch State Mines (DSM), a 4 billion dollar company. Products manufactured by Nipro were caprolactam nylon, Hi purity hydrogen, ammonium sulfate and sulfur dioxide. Columbia Nitrogen Corporation manufactured ammonia, nitric acid, ammonium nitrate, urea, and carbon dioxide. During my tenure, substantial cost reductions and plant

innovations were affected.

13. From 1978-1982, I was senior vice-president of Estech Corp., a subsidiary of Esmark, Inc. of Chicago, Illinois. There, my responsibilities included overseas operations in Australia, phosphoric acid, DAP and sulfuric acid productions, ammonia and ammonium nitrate production. I also oversaw the South Korean operations which produced substantially the same material. In the U.S., operations included two phosphate mines and ammonium, urea, nitric acid and ammonium nitrate facilities.

14. From 1983-1987, I was vice-president of engineering for Mid America Engineers, which included responsibilities for management of all engineering disciplines and project management of construction projects. Plant designs included printing plants, chemical plants, lubricating oil blending plants, toothpaste manufacturing facilities and soap manufacturing.

15. Between 1987-1994, I was executive vice-president for Vigoro Corporation of Chicago, Illinois. As executive vice-president, I had broad responsibilities including all manufacturing, environmental, R&D, as well as evaluation of acquisitions. During my tenure, a deep potassium chloride solution mine and recovery facility was built in Michigan for which I had oversight responsibilities. Other operational responsibilities included operation oversight a for nitrogen fertilizer manufacturer at East Dubuque, Illinois and in Canada, a large potassium chloride solution mining and production operation.

16. Between 1994 -1996, I was a consultant for Vigoro Corporation and visited various chemical plants which were candidates for potential purchase by the

company.

17. The processes on which I have worked over the years and in which I have had significant experience on the operational and design side includes ammunition and detonators, Dacron polyester (terylene) soda ash (solvay process), dense soda ash, bicarbonate of soda, calcium chloride and ammonium chloride, lime kilns, limestone and dolomite mining, aluminum chloride, fluorinated and chlorinated hydrocarbons (refrigerants), hydrofluoric acid, aluminum fluoride, ethylene, debutanized aromatic concentrates, ammonia, urea, nitric acid, ammonium nitrate, sulfuric acid, oleum (fuming sulfuric acid), phosphoric acid, super phosphoric acid, Di ammonium phosphate (DAP), super phosphates, caprolactam (nylon), purified hydrogen, ammonium sulfate, phosphate rock mining, potassium chloride and sodium chloride solution mining and potassium chloride recovery.

18. Based on my experience in chemical process design and chemical process operation, as well as my years of designing and building chemical plants, it is my opinion that one of ordinary skill in the art to which my invention is directed at the time the invention was made would require a chemical engineering degree at the Bachelor's level with between about 3-6 years of process design experience.

19. In the interview December 22, 1999, among Eileen E. Nave (Junior Examiner), Gary Straub (Primary Examiner), William Rigby (an inventor) and Harry Levy (attorney for inventors), Examiner Straub took the position that the Weber patent relied on by the Examiner was the principle and only reference used to reject the claims. Further, according to Examiner Straub, the Weber U.S. patent no. 1,500,289

disclosed the use of sulfur dioxide as opposed to sulfuric acid as argued by the inventor. The Examiner considered the statement in Weber on page 3, top right hand column, that roaster gas was used to provide sulfur dioxide to the reaction chamber. Examiner Straub took the position there is no chemical difference between the instant application and the Weber patent. The Examiners further considered the use of a fluidized bed to conduct the gas solid contact of Weber to be obvious in view of the expressed teaching in In re Edwards, a 1956 CCPA case.

20. The Weber patent clearly states that it is based on Hargrave's process. The Hargraves process is described on page 3 of the subject patent application and includes the teaching that sodium and potassium chloride can be converted to the sulfate salts and hydrochloric acid by contacting the chloride salts with sulfuric acid made by a process similar to the old chamber process involving oxidation of the sulfurous acid with air and water vapor. The discussion of the short comings of the Hargrave process occurs in the last paragraph of page 3 of the instant patent application and in the Weber '289 patent.

21. Primary Examiner Straub stated at the interview that the Weber patent discloses the use of  $\text{SO}_2$  and pointed to the reaction in column 1, line 29. It is submitted that Primary Examiner Straub misread the Weber '289 patent. The paragraph immediately preceding the reaction on line 29 relates to the reaction of sodium chloride with sulfurous acid in the presence of oxygen in steam. The Examiner in his reading of the Weber patent ignored the teaching of the Weber patent and focused only on the reaction. The reaction is a short hand method of referring to

sulfurous acid as specifically taught in the Weber patent. Based on my experience, the reaction which appears on line 21 of Weber is merely another way of expressing the chemical reaction of sodium chloride and sulfurous acid in the presence of oxygen and steam.

22. The Examiner is further directed to the discussion in the Weber patent at the left hand column on page 2, from line 50-line 80. There, Weber describes the contribution of the Hargraves process, but nowhere states that the chemistry suggested by Weber is any different than the chemistry disclosed in the Hargraves process.

23. Weber on page 3, lines 61-75, does state that gas is drawn from the roasting furnace and blown into the interior of the shaft where it contacts the briquets of sodium chloride. Nowhere in the Weber patent is there disclosed an independent source of water or steam other than the water vapor which may be contained in the ambient air. At the interview, Primary Examiner Straub took the position that the water in the air would be sufficient to drive the reaction as described and claimed in the instant patent application and that there was no difference between the chemical reaction of Weber and the chemical reaction of the instant process. Enclosed herewith as Exhibit A is a calculation assuming that the ambient air is at 75°F and is saturated with water. This is clearly not a condition which would frequently occur, but it is the most advantageous condition for the Examiner's position. The calculations show that in order to provide sufficient water for the reaction to proceed, 4-1/2 times the volume of air is required as compared to the inventive process to provide sufficient water to complete the conversion from potassium chloride to potassium sulfate. This excess air



would drastically reduce the temperatures of the gases exiting the roaster. In my opinion, such an excess of air would reduce the temperature of typical roaster gases to a temperature in the range of from about 250°F to about 300°F, at which temperature the reaction to produce potassium sulfide would not proceed. Weber does not teach the addition of sufficient water vapor to maintain the roaster gases at a temperature high enough to initiate the reaction of the sulfur values in the roaster gas and the potassium chloride briquets.

24. Even if the chemistries were identical, the use of sulfur dioxide, water vapor and oxygen with briquets would not replicate the invention. As stated at the interview, I have personally visited the plant in Dumas, Texas referred to on pages 5 and 6 of the instant patent application. In that plant, seven days were taken to transform briquets of potassium chloride to potassium sulfate and even then, the transformation was incomplete. As stated in the application, there was incomplete conversion at the interior of the briquets and over sulfation at the exterior of the briquets. Over sulfation is a problem because of the acidic properties of the over sulfated material. As stated at the interview, significant transportation problems occurred because the acidic nature of the over sulfated portion of the briquets cause severe corrosion in railroad cars. Another problem with the briquet process as practiced by Hargraves, Weber and the Dumas plant is the production of excessive fines. Excessive production of fines are undesirable because they present disposal problems and cannot be used in blended fertilizer packages because it is impossible to guarantee the composition of blended fertilizers containing fines due to the difficulty in

transporting fines through bins. All of this was discussed with both Examiners at the interview. At the time I visited the Dumas, Texas plant and until my invention, it is my understanding and belief that one of ordinary skill in this art assumed that the reaction between the sulfur containing gas and the potassium chloride took a substantial period of time, on the order of seven days. This, I believe is the reason that fluidized beds have never been suggested for this process from the time of Hargraves (1874), until the instant application. If a person of ordinary skill in the art believes that a chemical reaction requires on the order of seven days to complete, then it cannot be obvious, and in my opinion, it is not obvious to one of ordinary skill in this art, to use a fluidized bed reaction for a chemical reaction which takes days to complete.

25. During the interview, I discussed with the Examiners, the building of a potassium sulfate plant in Canada in the late 1980s. I told the Examiners that this plant was not based on fluidization but rather on ion exchange, a much more complicated process requiring more expensive equipment and energy to concentrate solutions and precipitate potassium sulfate and which ultimately became uneconomical.

26. Using a fluidized bed to effect the conversion of potassium chloride to potassium sulfate, a number of benefits occur, none of which would have been obvious to one of ordinary skill in the art to which the invention is directed at the time the invention was made because such a person would not have believed that the reaction could occur in a fluidized bed. These advantages are complete conversion of the potassium chloride to potassium sulfate, thereby enabling the use of the product as a fertilizer, even in such agricultural applications where chloride traces are impermissible.

The reaction is substantially instantaneous and the chemistry is substantially uniform across the particle. The potassium chloride particles in the fluidized bed retain substantially the same physical characteristics, both in particle size and particle size distribution. All of these advantages would not have been obvious to one of ordinary skill in the art to which the invention was directed at the time of the invention because a fluidized bed was thought to be inapplicable to the process.

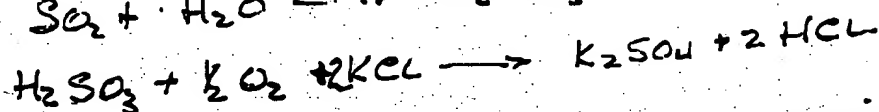
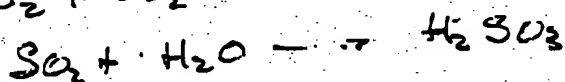
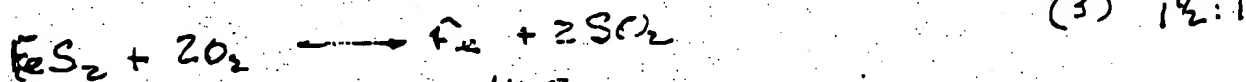
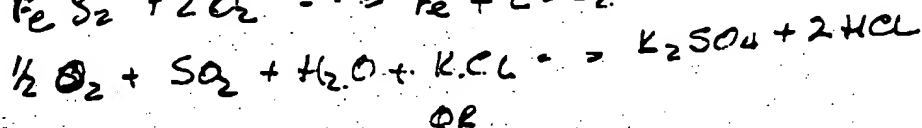
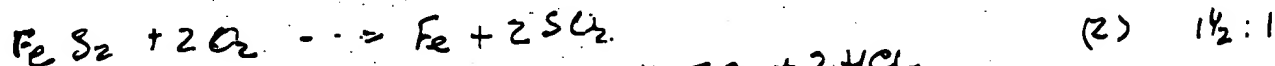
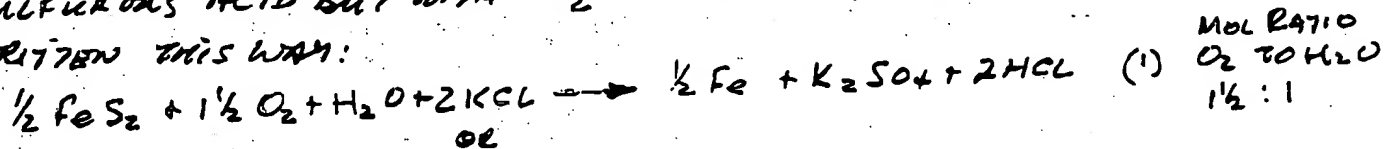
27. I declare further that all statements made in this declaration of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Further declarant sayeth not.

Dated: 1/13/2000

  
William J. Rigby

THE EXAMINER CLAIMS THAT IN WEBER THE REACTION IS NOT WITH SULFURIC ACID BUT WITH  $SO_2$ . IN REALITY THE REACTION MAY BE WRITTEN THIS WAY:



IN OTHER WORDS THE WAY THE REACTION IS WRITTEN MAY BEAR NO RELATION TO THE PROCESS THAT IS TAKING PLACE, THE FACT THAT WEBER WRITES THE REACTION SHOWING  $SO_2$  DOES NOT MEAN THEREFORE THAT THAT WAS THE PROCESS USED, PARTICULARLY SINCE

1. HE STATES THAT HE IS USING THE HARGREAVES PROCESS WHICH IS IN FACT BASED ON SULFUROUS ACID.
2. IN THE BODY OF THE PATENT HE STATES THAT HIS PROCESS IS BASED ON SULFUROUS ACID.
3. HIS CLAIM IS BASED ON SULFUROUS ACID.

WEBER DOES NOT EXPLAIN HOW HE OBTAINS THE GASEOUS ACID HIS DEPICTION OF THE PROCESS DOES NOT SHOW WHEN OR HOW WATER IS ADDED. IT COULD HAVE BEEN SUPPLIED AS SULFUROUS ACID FROM AN OLD CHAMBER PROCESS, WHICH IN TURN RECEIVED  $SO_2$  FROM PYRITE ROASTING.

THE EXAMINER CLAIMS THAT THERE IS SUFFICIENT WATER IN THE AIR TO ADEQUATELY SATISFY THE REACTION OF  $SO_2$ , OXYGEN & WATER. THE FOLLOWING CALCULATIONS SHOW THAT EXCESSIVE EXCESS AIR WOULD BE REQUIRED TO PROVIDE ADEQUATE WATER, WHICH WOULD SIGNIFICANTLY REDUCE GAS TEMPERATURES, SO THAT THE RX WOULD NOT INITIATE.

WEBER P. 2

CALCULATION OF AIR & H<sub>2</sub>O REQD.

EVERY MOL OF H<sub>2</sub>O REQUIRES 1 1/2 mols O<sub>2</sub>

Air Composition (Molecular)

N<sub>2</sub> 72%

O<sub>2</sub> 21%

Ar & OTHER 1%

Av. mol. wt of Air 29.0

Density of Air (75°F) 0.075 <sup>lbs/ft<sup>3</sup></sup>

Assume BEST CASE ~~max~~ i.e. max moisture available in Air saturated air at 75°F.

100% Humidity at 75°F sat. 0.1309 <sup>lb H<sub>2</sub>O / lb Dry Air</sup>

$$\text{or } \frac{130}{1000} = \boxed{0.0186 \frac{16 \text{ H}_2\text{O}}{16 \text{ Dry Air}}}$$

$$\text{mol. wt. H}_2\text{O} = 18 \frac{16}{16}$$

$$= \frac{0.0186}{18} \frac{16 \text{ H}_2\text{O}}{16 \text{ Dry Air}} \times \frac{16 \text{ H}_2\text{O}}{16 \text{ H}_2\text{O}} =$$

$$\boxed{0.0010 \frac{\text{mole H}_2\text{O}}{16 \text{ Dry Air}}}$$

Moles of O<sub>2</sub> / 16 Dry Air

$$\% \text{ mol. wt O}_2 \text{ in Air} = \frac{0.21}{1} \frac{\text{mole O}_2}{\text{mole Dry Air}}$$

$$\text{mol. wt Air} = 29.0$$

$$\frac{\text{Mole O}_2}{16 \text{ Dry Air}} = \frac{0.21}{29} = \boxed{0.0072 \frac{\text{mole O}_2}{16 \text{ Dry Air}}}$$

BASED ON REACTIONS SHOWN 1 1/2 mols O<sub>2</sub> & 1 mol of H<sub>2</sub>O are required to produce 1 mol K<sub>2</sub>SO<sub>4</sub>.

WEBER P. 3.

$$\frac{1.5}{0.0072}$$

$$\frac{\text{mole O}_2}{\text{mole O}_2}$$

$$= 205.33 \text{ lbs Dry Air}$$

Reqd to produce 1.5 mole O<sub>2</sub>

Req'd to provide 1.5 mole O<sub>2</sub>  
 & 1 mole K<sub>2</sub>SO<sub>4</sub>

&  $\frac{1}{0.001}$

mole H<sub>2</sub>O

1 mole H<sub>2</sub>O

16 Dry Air

1000 lbs Dry Air

Req'd to provide 1 mole H<sub>2</sub>O  
 & 1 mole K<sub>2</sub>SO<sub>4</sub>

1.4.

4.8 TIMES MORE SATURATED AIR AT 75°F ARE REQUIRED TO PROVIDE SUFFICIENT WATER TO COMPLETE CONVERSION OF KCL TO K<sub>2</sub>SO<sub>4</sub>. THAN ~~THE~~ THE AMOUNT REQUIRED TO PROVIDE THE OXYGEN FOR THE CONVERSION.

THIS EXCESS AIR WOULD DRASTICALLY REDUCE THE TEMPERATURE OF THE GASES EXITING THE ROASTER. I ESTIMATE IT WOULD REDUCE THE TEMPERATURE TO 250 TO 300°F AT WHICH TEMPERATURE THE REACTION WOULD NOT PROCEED.

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